

APPLICATION OF MISMATCH INDUCED SUPERPLASTICITY TO  
THE CONSOLIDATION OF COMPOSITE COMPACTS

AUTHOR:  
GLENN S. DAEHN  
DEPARTMENT OF MATERIALS SCIENCE AND ENGINEERING  
THE OHIO STATE UNIVERSITY

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DEPARTMENT OF MATERIALS SCIENCE AND ENGINEERING  
THE OHIO STATE UNIVERSITY  
2041 COLLEGE ROAD  
COLUMBUS, OH 43210

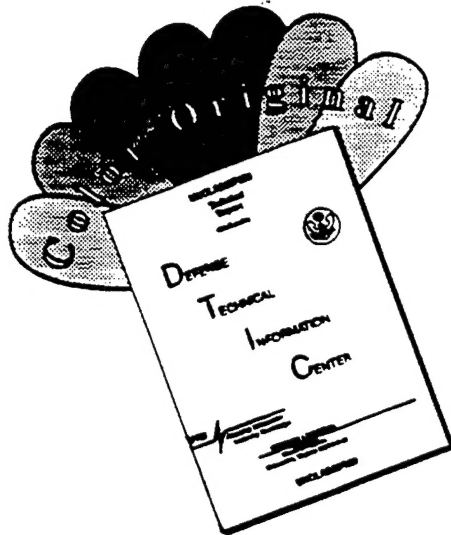
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| 13. ABSTRACT (Maximum 200 words)<br>It has been postulated that mismatch induced superplastic effects can be attained in the pressure cycling of materials that have a mismatch in compressibility. This project sought to: 1) experimentally verify that superplastic effects can be attained this way, and 2) apply this effect to the practical forming of metal matrix composites and the consolidation of composite powder compacts. This project was fully successful in demonstrating that the effect exists and can be applied to the practical compaction of composite powders. However, at present, the forming of composite sheets by pressure cycling does not appear to be practical (although has been demonstrated). Specifically it was shown pressure cycling can increase the density, density uniformity and strength (by factors up to 5) of green composite powder compacts by imposing proper pressure cycling. This is important as very minor modifications to conventional uniaxial (hot or cold) pressing equipment are all that are required to access these effects, strengths can increase strongly, and a simple model we have produced can guide the design of compaction processes. This may make possible the development of classes of highly reinforced composites that are presently impossible to produce. |  |   |   |  |
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### **Statement of the Problem:**

Superplastic effects have classically been seen in two modes; in the high temperature, modest strain-rate deformation of fine-grained materials (fine structure superplasticity) and in the thermal cycling of materials with mismatch in coefficients of thermal expansion (mismatch-induced superplasticity). It has been postulated that mismatch induced superplastic effects can also be attained in the pressure cycling of materials that have a mismatch in compressibility. This project sought to: 1) experimentally verify that superplastic effects can be attained this way and 2) apply this effect to the practical forming of metal matrix composites and the consolidation of composite powder compacts.

This project was fully successful in demonstrating that the effect exists and can be applied to the practical compaction of composite powders. However, at present, the forming of composite sheets by pressure cycling does not appear to be practical (although is theoretically possible).

### **Summary of Important Results:**

Superplasticity induced by thermal cycling has many advantages (it is not restricted to materials with fine grain sizes, for example), but large strains accumulate slowly. A thermal cycle of 300K in Al-SiC produces a strain near 0.5% per cycle. True strains of unity can thus be produced by 200 such cycles. Even under ideal conditions, for thin sections, several minutes are required for such forming. This rate typically renders the process unsuitable for mass production. Dunand notes that large phase transformation strains may be exploited at small thermal excursions, and thus conclude that this approach may be more practical for production. While large mismatch strains are attainable, the cycle time can be quite long because of heat transfer limitations in providing the necessary enthalpy of transformation. In contrast, stress or pressure cycle rates of more than 10Hz can be obtained routinely. This rapid pressure cycling has been the key concept in this program.

In the early part of this program we worked quite hard on trying to demonstrate superplasticity in Al-SiC metal matrix composites via imposing high temperature and a fixed force in one direction and a cyclic hydrostatic pressure. Via this approach we were able to demonstrate mismatch induced superplasticity via pressure cycling does work, as is demonstrated in publication 2, listed below. However, equipment issues (leaks, etc.) limited our progress and we focused on the effect of pressure cycling on powder consolidation for the remainder of the program.

Fine structure superplastic powders can be consolidated more effectively than those with low rate sensitivity (as shown by Sherby and Caliguri). An alternate route, following thermal cycling of MMC's, exploits mismatch plasticity to consolidate heterogeneous powders. The first demonstration of this concept was presented by Ruano and Sherby. They made use of the  $\alpha-\gamma$  phase transition in iron to aid powder consolidation, demonstrating nearly theoretical density of iron powders after 10 temperature cycles between 675°C and 775°C at a compaction stress of 30MPa. In comparison, after static compression for 30 minutes at 30MPa and 775°C, 2% porosity was still present.

Pressure changes (via compressibility differences,  $\Delta\beta$ ) induce Eshelby mismatch strains in much the same way as temperature changes (via thermal expansion coefficient differences,  $\Delta\alpha$ ). Pressure-induced strains are generally smaller than those produced by thermal expansion mismatch or phase transformation (at normal pressures and temperatures), but cycling rates can be much more rapid. The Eshelby "transformation strain" for isotropic components, whether pressure or temperature activated, is simply defined:

$$\epsilon_{ii}^T = \Delta\alpha\Delta T = \frac{1}{3}\Delta\beta\Delta P \quad (1)$$

where the second and third terms refer to temperature and pressure change respectively,  $\Delta\alpha$  or  $\Delta\beta$  are the differences between coefficients of thermal expansion or compressibilities of the two phases, and  $\Delta T$  or  $\Delta P$  are the driving variables, either temperature or pressure change. Fig. 1 illustrates the equivalence of the two terms. For the Al-SiC system, a  $\Delta T$  of 50K is approximately equivalent to a  $\Delta P$  of 200MPa (30ksi).

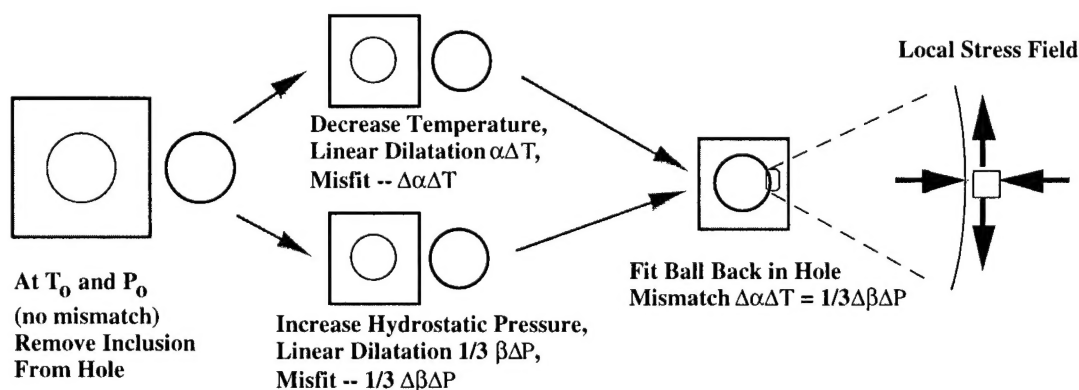


Fig. 1 - Equivalence of thermal and pressure mismatch strains

Our experiments presented in Papers 3-5 demonstrated that pressure cycling improves the consolidation of heterogeneous powders. As shown in Fig. 2, pressure cycling at room temperature increases the achievable density of Pb- $\text{Al}_2\text{O}_3$  powders, while having virtually no effect on homogeneous Pb powder. In agreement with our results and modeling for thermal cycling, the densification depends on the number of cycles and on the pressure amplitude, but it is independent of cycling frequency for rate-insensitive materials.

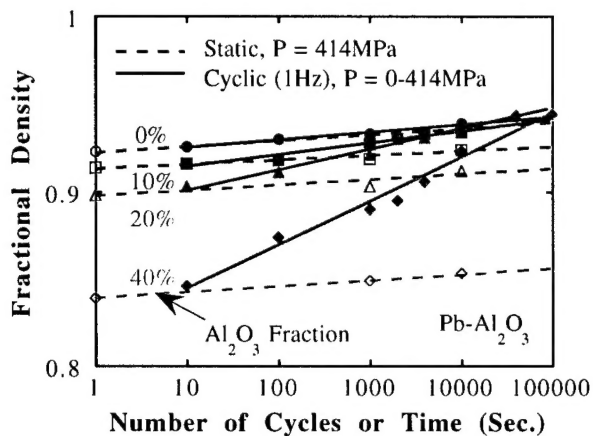


Fig. 2 - Fractional consolidation density for static and cyclic pressure conditions at room temperature (from Paper 3).

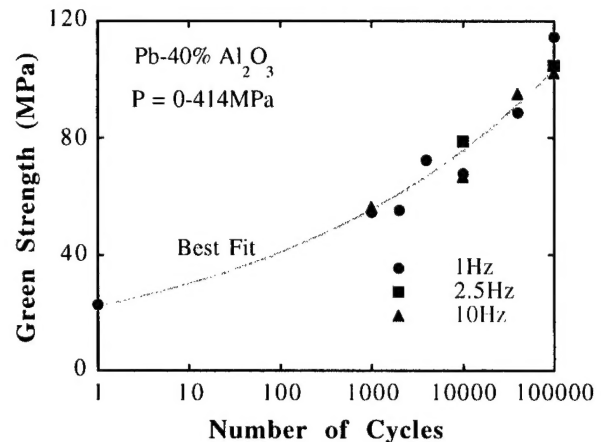


Fig. 3 - Development of green compact strength during pressure cycling [from paper 3].

The effect of pressure cycling on the green strength of such compacts is even more dramatic, Fig. 3. Whereas the density of pressure-cycled Pb-40%  $\text{Al}_2\text{O}_3$  is approximately 10% greater than for constant-pressure compaction (i.e., reducing porosity from 15% to 5%), the green strength increases by *more than 500%*. Thus, mismatch plasticity promotes the adhesion of powder particles. Micrographs (Fig. 4) confirm the improved degree of interconnection. Such compacts have the capability to withstand secondary processing, and thus create a new route for producing highly reinforced powder MMC's in systems which otherwise could not be processed.

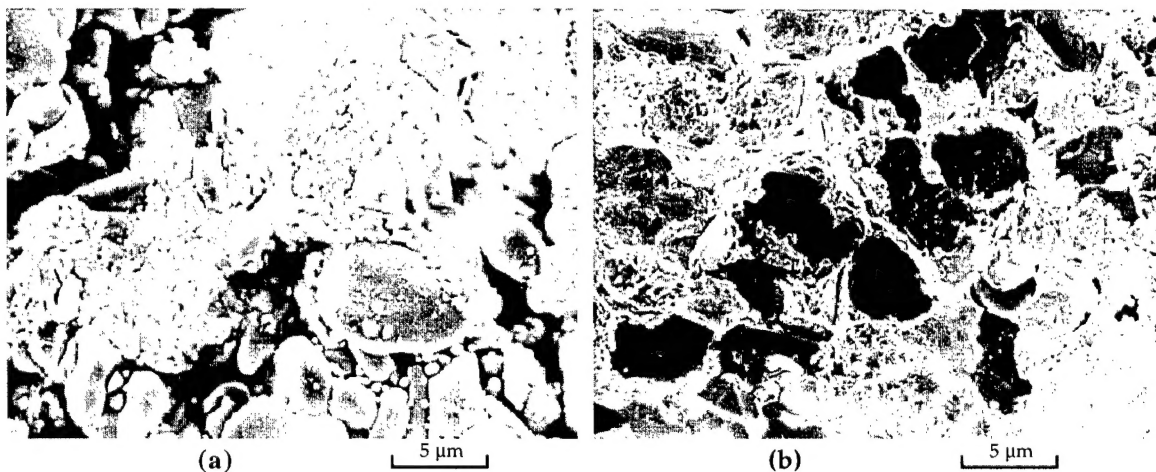


Fig. 4 - Fracture surfaces of Pb-40%  $\text{Al}_2\text{O}_3$  green compacts formed by static pressure of 414MPa (left) or  $10^5$  cycles of 0-414MPa (right) [from paper 3].

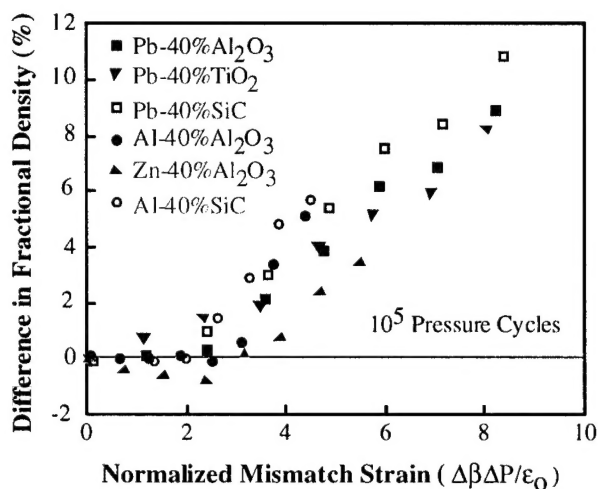


Fig. 5 - Difference in fractional density between the cyclic and static compaction as a function of the normalized mismatch strain [from paper 5].

dependence on the measure of elastic mismatch. Note that there is no improvement in systems and pressures corresponding to a value of  $\Delta\beta\Delta P/\epsilon_0$  less than approximately 3. In addition to the overall increase in density, the density uniformity is also dramatically increased by the application of pressure cycling. This is a key concept presented in Paper 4.

This fundamental work is quite important as very minor modifications to conventional uniaxial (hot or cold) pressing equipment are all that are required to access these effects, strengths can increase strongly and the simple model we have produced can guide the design of compaction processes. This may make possible the development of classes of highly reinforced composites that are presently impossible to produce.

#### Publications:

The following publications were a direct result of this contract:

1. "Yield of Continuous Fiber Composites Under Axisymmetric Loading", H. Zhang, S. Krishnaswami, P. M. Anderson and G. S. Daehn, *Scripta Metallurgica*, **29** 1477-84 (1993)
2. "Superplastic Forming of Metal Matrix Composites by Thermal and Pressure Cycling", C-Y Huang and G. S. Daehn, in *Superplasticity and Superplastic Forming*, A. K. Ghosh and T. R. Bieler, eds., TMS 1995, pp. 135-142.
3. "Densification of Composite Powder Compacts in Pressure Cycling", C-Y Huang and G. S. Daehn, *Acta Mater.*, **44**, 1035-45 (1996).
4. "Improved Densification and Uniformity of Composite Powder Compacts Due to Pressure Cycling", C-Y Huang and G. S. Daehn, in *Net Shape Processing of Powder Materials*, (edited by S. Krishnaswami, R. M. McMeeking and J. R. L. Trasserras), Vol. 216, p. 69, ASME, New York, NY (1995).
5. "Effect of Cyclic Pressure on the Low Temperature Consolidation of Several Composite

Other mechanisms can be postulated to account for improved powder densification with pressure cycling. (For example, modest improvements with brittle powders have been noted presumably related to packing rearrangement and particle cracking.) However, experiments for a wide range of heterogeneous systems and pressures suggest that the effect is directly related to normalized elastic mismatch strain between components,  $\Delta\beta\Delta P/\epsilon_0$ , where  $\epsilon_0$  is the more compressible material's yield strain. This is clearly shown in Paper 5. Fig. 5 reproduces these results and illustrates the universal

**Inventions:**

The concept of using pressure cycling to improve the density and strength of net-shape powder compacts was likely patentable. However, it was not pursued.

**Advanced Degrees:**

Ching-Yao Huang obtained a PhD degree based on work performed in this program. It was granted in June of 1996. He completed this degree in under four years at Ohio State. He is currently continuing as a Post-Doctoral researcher at Ohio State and seeking an assistant professorship.